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Indian Standard

SPECIFICATION FOR DELTAMETHRIN, TECHNICAL

UDC 632·951 DEL

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4

October 1987

Indian Standard

SPECIFICATION FOR DELTAMETHRIN, TECHNICAL

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(*Continued on page* 13)

Indian Standard

SPECIFICATION FOR DELTAMETHRIN, TECHNICAL

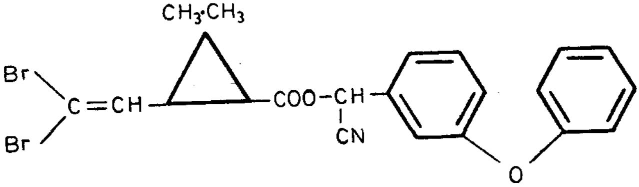
0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 29 May 1987, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Deltamethrin is an insecticide used in the control of pests of agricultural crops.

0.3 Deltamethrin is the accepted common name by the International Organization for Standardization (IS0) for (S) — α — Cyano — *m* — **phenoxybenzyl** (IR, 3R) — 3 -3 (2, 2-dibromovinyl) — 2, 2 dimethyl — Cyolopropane carboxylate. IUPC name of the product is (S) — a — cyan0 — 3 — phenoxybenzyl (IR) — *cis* — 3 (2, 2-dibromovinyl) — 2, 2-dimethyl cyolopropane carboxylate. The empirical and structural formulae, and the molecular mass are as given below:

Empirical Formulae	*Structural Formulae*	*Molecular Mass*
$C_{22}H_{19}Br_2NO_3$		*505.2*



0.4 In the preparation of this standard, due consideration has been given to the provisions of the ***Insecticides Act, 1968*** and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under the Act and Rules, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements, and the methods of sampling and test for deltamethrin, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of colourless to creamish coloured powder, odourless, and free from foreign matter and added modifying agents.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR DELTAMETHRIN, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix of this Standard	Cl No. of IS : 6940-1982*
(1)	(2)	(3)	(4)	(5)
i)	Deltamethrin content, percent by mass, *Min*	98·0	A	—
ii)	Melting point, °C	98·0-101·0	—	6
iii)	Optical rotation of 1 percent solution in benzene at 20°C	57 ± 1’5”	B	—
iv)	Acid chloride corresponding to deltamethrin, percent by mass, Max	0·2	C	—
v)	Acid + anhydride corresponding to deltamethrin, percent by mass, *Max*	1·0	D	—

*Methods of test for pesticides and their formulations (*first revision*).

3. PACKING AND MARKING

3.1 The material shall be packed according to the requirements given in IS : 8190 (Part 1)-1980*.

3.2 **Marking** — The container shall bear legibly and indelibly the following information and any other information as is necessary under the *Insecticides Act* and Rules:

a) Name of the material;

b) Name of the manufacturer or trade-mark;

---

*Requirements for packing of pesticides: Part 1 Solid pesticides *(first revision)*.

c) Date of manufacture;
d) Batch number;
e) Deltamethrin content, percent (m/m);
f) Net mass of the contents; and
g) The cautionary notice as worded in the *Insecticides Act* and Rules.

3.2.1 The container may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in IS : 10946-1984*.

5. TESTS

5.1 Tests shall be carried out as referred to in col 4 and 5 of Table 1.

5.2 **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF DELTAMETHRIN CONTENT

A-0. GENERAL

A-0.1 Either of the two methods, namely, HPLC method (see **A-1**) or total bromine analysis method (see A-2) shall be used for estimating deltamethrin content. However, HPLC method will be the referee method in case of dispute.

---

*Methods of sampling for technical grade pesticides.

†Specification for water for general laboratory use (*second revision*).

A-1. HPLC METHOD

A-1.1 Principle — After dilution of the sample, deltamethrin content is determined by comparing the response of the sample with that of a deltamethrin standard of known purity by HPLC on a column packed with silica.

A-1.2 Apparatus

A-1.2.1 ***Liquid Chromatograph*** — A suitable instrument for use with stainless steel columns and capable of maintaining pressures of 150 bars, fitted with a 20 microlitre loop injector.

A-1.2.2 ***Detector*** — UV spectrophotometer capable to measure UV absorption at 254 nm. Check its linearity in the concentration zone used for the determination.

A-1.2.3 ***Liquid Chromatographic Column*** — Stainless steel, 15-18 cm long, 4·6 mm internal diameter packed with Lichrosorb silica 60-80 mesh.

A-1.2.4 ***Recorder*** — One mV full scale recorder.

A-1.3 Reagents

A-1.3.1 ***Deltamethrin Standard*** — Of known purity.

A-1.3.2 ***Dioxan*** — UV spectroscopic grade, free of peroxides. Dioxan is mixed with 15 ml of water per litre.

A-1.3.3 ***Iso-octane*** — UV spectroscopic grade.

A-1.3.4 ***HPLC Mobile Phase*** — Mixture of dioxan and iso-octane in the ratio of 50 ml/950 ml.

A-1.4 Procedure

A-1.4.1 ***Preparation of the Standard Solution*** — Weigh, in duplicate, to the nearest mg approximately 50 mg of deltamethrin standard (r_1g and r_2g) into 50-ml volumetric flasks. Add a mixture of dioxan and iso-octane in the ratio of 200 ml/800 ml and shake the flasks to ensure dissolution of the standards. Dilute to volume with the same mixture (***Solutions*** R_1 and R_2).

A-1.4.2 Sample ***Preparation*** — Weigh in duplicate to the nearest 0·1 mg samples containing approximately 50 mg of deltamethrin (M_1g and M_2g) into 50-ml volumetric flasks. Add a mixture of dioxan and iso-octane in the ratio of 200 ml/800 ml and shake the flasks to dissolve the samples. Dilute to volume with the same mixture (***Solutions*** S_1 and S_2).

A-1.4.3 ***Liquid Chromatography Conditions*** — The conditions given below are typical. Conditions may have to be adjusted to obtain optimum results from a given apparatus provided standardization is done:

Column temperature	: Ambient
Flow rate	: 1 to 1·7 ml/min
Detector set at	: 254 nm
Approximate retention time	: 8 min

A-1.4.4 ***Procedure*** — With a 20-microlitre loop type injector, inject one of the standard solutions until peak height or area of two successive injections agree to within 2 percent. Inject the standard and sample solutions in succession according to the following sequences:

R_1S_1, R_2S_1, R_1S_2 and R_2S_2

A-1.4.5 ***Calculation***

$$\text{Deltamethrin content, percent by mass} = \frac{A_x \times r \times P}{A_r \times m}$$

where

A_x = deltamethrin peak area of the sample solution (S_1 or S_2);

r = mass, in g, of the deltamethrin in the standard solution (R_1 or R_2);

P = purity of deltamethrin standard;

A_r = deltamethrin peak area of the standard solution (R_1 or R_2); and

m = mass, in g, of the sample in the sample solution (S_1 or S_2).

NOTE — **Alternatively, the calculation can be based on the peak heights. The four results should agree to within ±3 percent of their mean value. If not, repeat the analysis.**

A-2. TOTAL BROMINE ANALYSIS METHOD

A-2.1 Reagents

A-2.1.1 *Isopropanol* — **pure.**

A-2.1.2 ***Metallic Sodium***

A-2.1.3 Formaldehyde

A-2.1.4 Phenolphthalein Solution — One percent (*m/m*) in rectified **Spirit.**

A-2.1.5 ***Nitric Acid***

A-2.1.6 *Benzene*

A-2.1.7 Silver Nitrate Solution — 0·1 N.

A-2.1.8 *Ammonium Thiocyanate Solution* — 0·1 N.

A-2.1.9 *Ferric Ammonium Sulphate Indicator Solution — Two percent* (*m/m*).

A-2.1.10 Nitrobenzene

A-2.2 **Procedure**

A-2.2.1 Weigh accurately a quantity of the material containing about 0·15 g of deltamethrin and transfer it to a 250-ml conical flask with standard joint. Add 5 ml of benzene and then 30 ml of iso-propanol, shake to mix. Add 2·5 g of freshly cut sodium metal and reflux it on a very low flame cautiously, using a wire gauze for about 2 hours. Then destroy the excess sodium metal by carefully adding 50 percent aqueous iso-propanol solution through the condenser at the rate of one or two drops per second. Boil again for 10 minutes. Stop heating, cool to room temperature. Add 20 ml water and shake. Then add 4 ml of formaldehyde, shake and keep for 10 minutes.

A-2.2.2 Add 1 or 2 drops of phenolphthalein. Acidify the solution with 50 percent nitric acid till the reversal of phenolphthalein indicator. Shake well.

A-2.2.3 Add 25 ml of silver nitrate, 2 ml of nitrobenzene and 5 ml of ferric ammonium sulphate indicator. Shake well.

A-2.2.4 Back titrate the excess of silver nitrate with ammonium thiocyanate solution to brick red colour.

A-2.2.5 Carry out a blank titration by exactly proceeding according to A-2.2.1 to A-2.2.4.

A-2.3 **Calculation**

$$\text{Deltamethrin content, percent by mass} = \frac{V \times 0{\cdot}025\,25 \times N \times 100}{M \times 0{\cdot}1}$$

where

V = volume, in ml, of 0·1 N ammonium thiocyanate consumed by deltamethrin;

N = normality of ammonium thiocyanate solution; and

M = mass, in g, of the material taken for the test.

APPENDIX B

[*Table* 1, Item (iii)]

DETERMINATION OF OPTICAL ROTATION

B-1. APPARATUS-A polarimeter on which angular rotation accurate to 0·05° can be read.

B-1.1 D-Line — Appropriate arrangement to obtain D-line of sodium or 546·1 nm line of mercury spectrum.

B-1.2 **Polarimeter Tubes** — Suitable polarimeter tube of 1 decimetre length.

B-2. PROCEDURE

B-2.1 **Sample Preparation** — Weigh accurately 1 g of deltamethrin and dissolve in benzene solvent. Make to 100 ml with benzene. Maintain the temperature of this solution at 20°C.

B-2.2 Pour benzene solvent maintained at 20°C into the dry polarimeter tube and close the end caps tightly so as to ensure that there are no air bubbles. Measure the optical rotation. This is the blank reading.

B-2.3 Pour the sample obtained in B-2.1 into a dry polarimeter tube and close the end caps-tightly, so as to ensure that there are no air bubbles. Measure the optical rotation. This is the sample reading.

B-3. CALCULATION

$$\text{Specific rotation} = \frac{a \times 100}{l \times c}$$

where

a = corrected observed rotation in degrees;

l = length of polarimeter tube in decimetre; and

c = concentration of solution expressed in g of the substance in 100 ml solution.

APPENDIX C

[*Table* **1,** *Item* (iv)]

DETERMINATION OF ACID CHLORIDE CORRESPONDING TO DELTAMETHRIN

C-1. REAGENTS

C-1.1 Methanolic Potassium Hydroxide Solution — 0·02 N.

C-1.2 Neutralized Methanol — To 100 ml of methanol, add 3 drops of a 1 percent solution of bromophenol blue in methanol. If necessary, add 0·1 N solution of hydrochloric acid in methanol (obtained by diluting 8 ml of 12 N hydrochloric acid to 100 ml with methanol) until yellow colour develops. Add 0·02 methanolic potassium hydroxide until a purple blue colour develops.

C-2. PROCEDURE

C-2.1 Transfer a sample of about 2 g of product weighed to the nearest 0·1 mg into a 200-ml Erlenmeyer flask. Add 100 ml of neutralized methanol. Warm to dissolve and let cool to room temperature. Allow to stand for 5 min and titrate with the standard 0·02 N methanolic potassium hydroxide solution to purple blue end-point.

C-2.3 **Calculation**

Milliequivalent for 1 g corresponding to the acid chloride $B = \frac{t \times N}{m}$

where

t = volume, in ml, of standard methanolic potassium hydroxide used;

N = normality of the methanolic potassium hydroxide; and

m = mass, in g, of the sample taken for the test.

Acid chloride content, percent by mass $= \frac{B \times 316.45 \times 100}{1\ 000}$

$= B \times 31.645$

APPENDIX D

[*Table* 1, Item (v)]

DETERMINATION OF CONTENT OF ACID + ANHYDRIDE CORRESPONDING TO DELTAMETHRIN

D-1. DETERMINATION OF ACID

D-1.1 Reagents

D-1.1.1 ***Ethanolic Sodium Hydroxide Solution*** — 0·02.

D-1.1.2 ***Neutralized Ethanol*** — To 100 ml of ethanol, add 10 drops of a 1 percent solution of alphanaphtholbenzene in ethanol. Add 0·02 N ethanolic sodium hydroxide until a true green colour develops.

D-1.2 Procedure — Transfer a sample of about 2 g of product weighed to the nearest 0·1 mg into a 200-ml Erlenmeyer flask. Add 100 ml of neutralized ethanol. Warm to dissolve, cool in ice and titrate immediately the ice-cooled solution with the standard 0·02 N ethanolic sodium hydroxide solution to a true green end-point.

D-1.3 Calculation

Milliequivalent for 1 g corresponding to acid + acid chloride

$$c = \frac{t_1 \times N}{m_1}$$

where

t_1 = volume, in ml, of standard ethanolic sodium hydroxide used;

N = normality of the ethanolic sodium hydroxide; and

m_1 = mass, in g, of the sample taken for the test.

Acid content, percent by mass = $\frac{(C - B) \times 297{\cdot}95 \times 100}{1\,000} = (C - B) \times 29{\cdot}795$

D-2. DETERMINATION OF ANHYDRIDE

D-2.1 Reagents

D-2.1.1 ***Aniline in Cyclohexane*** — 0·1 N.

D-2.1.2 ***Perchloric Acid Solution in Glacial Acetic Acid*** — 0·1 N. Mix 70 to 72 percent perchloric acid carefully with glacial anhydrous acetic acid (about 500 ml) and pure acetic anhydride (50 ml) and make up to one litre with acetic acid. ***The flask shall be well cooled while the reagents are being mixed.***

D-2.1.2.1 *Standardization* — Weigh accurately about 0·02 g of anhydrous sodium carbonate and dissolve it in acetic acid (30 ml). Add 1-naphtholbenzene indicator 1 percent solution in benzene (2 drops) and titrate with perchloric acid until the colour of the solution changes from yellow orange to dark green.

$$\text{Normality } (N) = \frac{1\,000\, m}{52{\cdot}994\, t}$$

$$\frac{18{\cdot}87\, m}{t}$$

where

m = mass of anhydrous sodium carbonate; and

t = volume of perchloric acid used.

NOTE — When titrating with perchloric acid in glacial acetic acid, the following precautions shall be observed:

a) There should be complete absence of water. If water is present, no satisfactory end point will be obtained.

b) Titrations and standardizations should be carried out at the same temperature because acetic acid has a large coefficient of thermal expansion.

D-2.2 Procedure — Transfer a sample of about 1 g of product weighed to the nearest 0·1 mg into a 200-ml Erlenmeyer flask. Add 10 ml accurately measured 0·01 N aniline solution in cyclohexane and 10 ml of glacial acetic acid. Stopper the flask, mix and allow to stand for 1 hour at 20-25°C. Titrate with 0·01 N perchloric acid in the presence of 1 percent solution of crystal violet in acetic acid until the initially purple colour turns emerald green.

D-2.2.1 Run a blank test in the same conditions but omitting the sample.

D-2.3 Calculation

Milliequivalent for 1 g corresponding to anhydride and two times acid chloride

$$D = \frac{(T - t_2) \times N}{m_2}$$

where

T = volume, in ml, of 0·01 N perchloric acid used in the titration for the sample;

t_2 = volume, in ml, of 0·01 N perchloric acid used for the blank;

N = normality of the perchloric acid used; and

m_2 = mass, in g, of the sample taken for the test.

Anhydride content, percent by mass

$$= \frac{(D - 2B) \times 577{\cdot}9 \times 100}{1\,000} = (D - 2B) \times 57.79$$

(Continued from page 2)

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 w = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	HZ	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

AMENDMENT NO. 1 MAY 2002
TO
IS 12005 : 1987 SPECIFICATION FOR DELTAMETHRIN, TECHNICAL

(*Page* 4, *Table* 1, *Sl No.* 3, *col* 2) — Substitute '4 percent' *for* '1 percent' and 'toluene' *for* 'benzene'.

(*Page* 8, *clause* **A-2.1.6**) — Substitute 'Toluene' *for* 'Benzene'.

(*Page* 8, *clause* **A-2.2.1**, *line* 3) — Substitute 'toluene' *for* 'benzene'.

(*Page* 9, *clause* **B-2.1**) — Substitute '4 g' *for* '1 g' and 'toluene' *for* 'benzene' .

(*Page* 9, *clause* **B-2.2**, *line* 1) — Substitute 'toluene' *for* 'benzene'.

(FAD 1)

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